PATENT
Attorney Docket No. 02019CON
LVM Reference No. 225308

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Kutsovsky

Application No. 10/720,582

Art Unit: 1793

Examiner: Wartalowicz, Paul A.

Filed: November 24, 2003

For: FUMED METAL OXIDE PARTICLES AND PROCESS FOR PRODUCING THE SAME

DECLARATION UNDER 37 C.F.R. § 1.132 OF GAEL D. ULRICH

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

I, Gael D. Ulrich, hereby declare that:

1. I have been a consultant in the field of chemical engineering and process design for a number of corporations, including Cabot Corporation, which is the assignee of the subject patent application. My specialty is combustion with particular expertise in the theory and practice of particle formation and growth in combustion processes. This includes flame-generated fine particle chemicals, welding fumes, and fly ash produced by the burning of coal, oil, and trash. I have more than 40 years of experience in this area.

2. I received Bachelor of Science and Masters of Science degrees in chemical engineering from the University of Utah in 1959 and 1962, respectively. I received my doctoral degree in chemical engineering from the Massachusetts Institute of Technology (MIT) in 1964. After obtaining my doctoral degree, I held the position of Senior Research Engineer at Atomics International (Northridge, California) from 1964 to 1965, and Senior Research Engineer at Cabot Corporation (Billerica, Massachusetts) from 1965 to 1970. I left Cabot to take a teaching

position at the University of New Hampshire until my semi-retirement in 1998. I am currently Professor Emeritus of Chemical Engineering at the University of New Hampshire.

3. I have written or co-authored numerous technical journal articles and three books. My best-known journal article was a Special Report on *Flame-Generated Fine Particles* that was featured as the cover piece in the August 4, 1984, issue of *Chemical and Engineering News*. Interest from industrial readers of that article prompted me to form Ulrich Research and Consulting, Inc. From 1985 to 1995, I, in concert with three other engineers, performed proprietary research for commercial clients on flame-generated fine particle chemicals.

4. In more recent years, I have written or co-authored articles in *Chemical Engineering* (March 2006, April 2006, and September 2006, as well as an article scheduled for publication in April 2009) and *Chemical Engineering Progress* (July 2006, and June 2007). These articles pertain to fundamental chemical engineering process design and economics topics based on various chapters of my third book, *Chemical Engineering Process Design and Economics, A Practical Guide* (2nd edition, Process Publishing, 2004). This was a revision of my first book, *A Guide to Chemical Engineering Process Design and Economics* published by John Wiley and Sons in 1984. Process design and economics was a course that I taught every year at the University of New Hampshire. *A Guide to Chemical Engineering Reactor Design and Kinetics* was another textbook (published privately, Ulrich Publishing, 1994) developed for another course that I taught almost every year at University of New Hampshire.

5. My research both in industry and academia was focused on the formation and growth of particles in flames, primarily various metal oxides. I had other projects as well dealing with fly ash from coal combustion, and welding fume. I believe that most people familiar with the literature on particle formation in high temperature systems would consider me a pioneer and a respected authority on the subject. One prominent researcher, in citing my work, called me a "visionary." (Let's assume he intended the positive connotation of that term.)

6. I understand that the subject patent application claims a process for preparing fumed metal oxide particles involving the post-flame injection of a liquid feedstock into a stream

of combusted gas such that the liquid feedstock is atomized and then burned or pyrolyzed to convert the liquid feedstock into fumed metal oxide particles. In particular, I understand that the subject patent application claims a process for preparing fumed metal oxide particles, wherein the process includes the steps of (a) delivering one or more streams of an oxidant and a liquid or gaseous fuel to a combustion chamber of a reactor, (b) combusting the oxidant and liquid or gaseous fuel in the combustion chamber of the reactor, thereby forming a stream of combustion gas which flows out of the combustion chamber, (c) providing a stream of a liquid feedstock comprising a volatizable non-halogenated metal oxide precursor and injecting the stream of the liquid feedstock into the stream of combustion gas to thereby atomize the liquid feedstock within the stream of combustion gas and form a reaction mixture comprising the combustion gas and the atomized liquid feedstock, and (d) subjecting the atomized liquid feedstock to a sufficient temperature and residence time in the stream of combustion gas for the liquid feedstock to combust or pyrolyze and thereby be converted to fumed metal oxide particles, which are subsequently recovered.

7. I understand that the following references have been cited by the Examiner at the U.S. Patent and Trademark Office during the prosecution of the subject patent application:

U.S. Patent 5,340,560 (Rohr et al.),
U.S. Patent 5,256,389 (Jordan et al.),
U.S. Patent 6,312,656 (Blackwell et al.),
U.S. Patent 5,904,762 (Mahmud et al.),
U.S. Patent 4,822,410 (Matovich),
U.S. Patent 5,075,090 (Lewis et al.),
U.S. Patent 6,565,823 (Hawtof et al.), and
U.S. Patent 4,857,076 (Pearson et al.).

I have reviewed each of the aforementioned references.

8. Other than the Rohr '560 patent, none of the other cited references applies to the production of fumed metal oxides. Fumed metal oxides have a unique physical structure. In

particular, they usually consist of multiple substantially spherical primary particles fused together in a chain-like aggregate structure. In contrast, precipitated metal oxides particles, condensation-polymerized metal oxide particles, pigmentary oxides, and the like are generally discrete spherical or crystalline particles.

9. For example, the Jordan '389 patent describes the preparation of foamed metal oxide particles by injecting a decomposable metal salt solution into a flame. As depicted by transmission electron micrographs (TEMs) and scanning electron micrographs (SEMs) of the resulting particles (see Figures 3a-5b of the Jordan '389 patent), the produced foamed metal oxide is clearly not the same as fumed metal oxide. Evidently, the solvent evaporates to leave a viscous salt residue that boils internally to create a foam or sponge.

10. The Blackwell '656 patent describes the preparation of what the author calls "amorphous soot" by injecting a liquid precursor into a flame that is trained on a mandrel. This forms a large ceramic body that is eventually placed in a furnace from which optical fibers are drawn. The particle structure of the amorphous soot is different from that of fumed metal oxides.

11. The Hawtof '823 patent is almost identical to Blackwell '656. Again, amorphous metal oxide is not the same as fumed metal oxide.

12. The Mahmud '762 patent describes the preparation of dual-phase carbon/silica particles involving the post-flame injection of a liquid precursor. The resulting dual-phase carbon/silica is not the same as fumed metal oxide.

13. The Matovich '410 patent describes a high-temperature chemical reaction process for the reclamation of metals, essentially a smelting operation. There is no similarity between the resultant metallic and/or refractory beads and fumed metal oxides.

14. The Lewis '090 patent describes a process for preparing what the authors call "calcined" metal oxide particles for making ceramic bodies. I would use the term "tempered." In essence they use a conventional premixed flame but subject the product to longer residence times and higher temperatures to create large single particles and crystallites—forms that serve

better for making ceramic bodies. I see no significant similarities between this and the process claimed in the subject patent application – either as to process steps or the resulting product.

15. The Pearson '076 patent describes a process for preparing synthesis gas containing hydrogen and carbon monoxide by the partial oxidation of a slurry of solid carbonaceous fuel in a liquid carrier. Products of combustion are all gases—not particles. I see no similarity with the process claimed in the subject patent application.

16. As indicated above, fumed metal oxide particles differ from foamed particles, amorphous soot, dual-phased carbon/silica particles, and gases. I find nothing in the cited references that suggests high quality fumed metal oxides could be made by injecting a liquid feedstock into a stream of combusted gas.

17. The Rohr '560 patent describes a conventional process for preparing fumed metal oxide particles. That is, the liquid feedstock is vaporized before mixing with fuel and oxidant and before combustion. It's a premixed flame of the type used traditionally to make fumed metal oxides.

18. The process claimed in the subject patent application differs from the conventional process described in the Rohr '560 patent in at least two ways, namely (i) the feedstock is in liquid (rather than gaseous) form when injected into the process stream and (ii) the process stream itself is post-flame rather than pre-flame. The cited references do not provide any reason to modify the conventional process in the manner necessary to provide the process claimed in the subject patent application.

19. Of the patents mentioned above, the process described in the Mahmud '762 patent is most similar to the process claimed in the subject patent application in terms of reagent addition and combustion sequence. However, the process described in the Mahmud '762 patent creates particles of mixed carbon and silica phases, which is quite a different product than the fumed metal oxides produced by the process claimed in the subject patent application. The contrast leads interestingly to some general differences between carbon black- and silica-

synthesis flames. In general, people making carbon black want to form particles and then freeze them quickly. If reactions are allowed to go to equilibrium, carbon black particles will oxidize further or burn away, thereby resulting in reduced yield. The opposite is true of fumed metal oxides. Chemical equilibrium is the most desired state in producing fumed metal oxides because yield is highest.

20. As indicated above, I have been involved in this field for over 40 years. I have tried on occasion to develop metal oxide burners having higher combustion intensity or other characteristics that might lead to unusual products or higher throughput. My experience has been that any change in a fumed metal oxide flame that inhibits progress toward equilibrium – such as described for carbon black production in the Mahmud '762 patent – has a negative effect on product quality. That is, any hint of unreacted feedstock is reflected by poorer product performance. Thus, injecting liquid feedstock into the downstream flame plume as described in the subject patent application would not have been promising in my opinion.

21. I hereby declare that I believe all statements made herein true with the knowledge that willful false statements are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: 1 April 2009

Gael D. Ulrich